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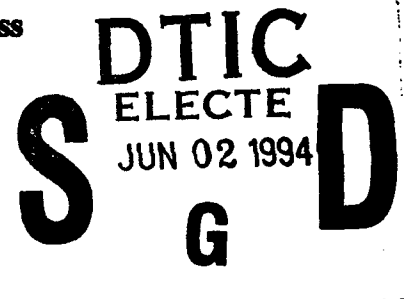
**Technical Report #15**

***A Low Temperature, Ultrahigh Vacuum, Microwave-Frequency-Compatible Scanning Tunneling Microscope***

by

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# **A Low Temperature, Ultrahigh Vacuum, Microwave-Frequency-Compatible Scanning Tunneling Microscope**

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## **Abstract**

To expand the capabilities of the microwave frequency alternating current scanning tunneling microscope to include the ability to study isolated adsorbates and highly reactive surfaces, we have developed a low temperature, ultrahigh vacuum alternating current scanning tunneling microscope. In this alternating current scanning tunneling microscope, we employ the reliable beetle-style sample approach mechanism with a number of other components unique to a low temperature scanning tunneling microscope. These include the sample transfer, delivery, retrieval, storage, sputtering, and heating systems. This alternating current scanning tunneling microscope has been operated at 77K and 4K.

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## I. Introduction

The ability to prepare and to study stable surfaces of known composition free of contamination has become a requisite in the development of the spectroscopic capabilities of alternating current scanning tunneling microscopy (ACSTM). With the tunability of our ACSTM design,<sup>1,2</sup> we are able to perform experiments where spectroscopic contrast between different surface sites is evident.<sup>3</sup> In order to understand the nature of this contrast we need to prepare and to study well characterized model systems. A model system could be an isolated adsorbate on a clean single crystal surface. To study such a model system we have designed and operated a tunable ACSTM in ultrahigh vacuum (UHV) and at low temperature (LT). Operation in UHV also provides us with the ability to perform standard surface analyses to complement our ACSTM observations. By transferring samples between the room temperature sample preparation/analysis chamber and the load lock, we are able to prepare, control, and characterize our sample surfaces. Samples can then be transferred to the low temperature ACSTM chamber which is kept at cryogenic temperatures (4K or 77K).

The UHV capabilities of this instrument include multiple sample transport, load lock sample entry, ion sputtering, mass spectroscopy, radiative and electron beam heating, and both conventional and AC scanning tunneling microscopy. Two ACSTMs are in the ultrahigh vacuum chambers. One ACSTM operates at room temperature and is used for sample characterization prior to transfer to the low temperature stage. The other ACSTM is in a chamber held at cryogenic temperatures down to 4.2K. Both ACSTMs can function as conventional (DC) STMs. In addition, each STM can operate simultaneously at conventional and microwave frequencies. As in our previous ACSTM design,<sup>4</sup> our detection scheme uses extremely sensitive microwave communications equipment.<sup>1,2</sup> This provides us with broad range tunability from DC–20 GHz.

In this paper we discuss the ACSTM design, sample transport, and sample preparation facilities specifically developed for beetle-style sample holders and microscopes.

## II. Instrument Design

### A. Instrument Layout

A schematic of the system layout is shown in Fig. 1. The vertical translator is mounted on top of a 0.25m spherical UHV chamber. This translator is used to move the sample from the surface preparation/analysis level of the chamber to the low temperature ACSTM elevator located 1.4m below. The vertical translator is also used to place the sample holder at the various surface preparation and analysis stages as shown in Fig. 2. The horizontal translator carries the sample holders to and from the load lock, the room temperature ACSTM, and the surface analysis/preparation chamber. Mirrors are placed on the ends of the horizontal translator and sample storage tray, see Fig. 2. These provide views of the sample holders in position on the ACSTMs. In this way we can monitor the orientation of the sample holder as it rotates on the beetle tripod during sample approach.<sup>5</sup>

The low temperature ACSTM is housed at the bottom of a cryogenic UHV chamber. Our cryogenic UHV chamber is based on the design of Eigler.<sup>6</sup> The cryogenic chamber is cooled by submersion in liquid helium or liquid nitrogen. The ACSTM is cooled by OFHC Cu thermal links to the UHV chamber walls which are in contact with the liquid cryogen. All UHV electrical and mechanical feedthroughs are standard components. The microwave feedthroughs are double-ended SMA connectors mounted on mini-conflat flanges.<sup>7</sup> The two stages — cryogenic and sample preparation — are mounted on separate platforms and are vibrationally decoupled by an interconnecting welded bellows. The cryogenic and sample preparation platforms are each isolated from the main vibration isolation platform (a custom laser table with pneumatic legs)<sup>8</sup> by air-filled pneumatic vibration dampers.<sup>9</sup> All non-UHV components are coated with epoxy paint to reduce acoustic pickup and ringing/resonating. Further reduction of ringing/resonating was accomplished by placing mastic material on exposed components.<sup>10</sup> This decouples the ACSTMs from the rest of the system well enough that no additional isolation at the STM stage is needed. The turbomolecular pump and its associated backing pump are vibrationally decoupled from the chamber using a

commercially available vibration isolator<sup>11</sup> and a bellows foreline encased by sand. Despite being rigidly mounted to and not otherwise vibrationally isolated from their respective chambers, both ACSTMs have obtained atomic resolution features on a variety of surfaces. Further, no sharp resonances are apparent in the DC tunneling current up to 20 kHz.

## B. AC Scanning Tunneling Microscope

The beetle style ACSTM microwave design and operation have been reported elsewhere.<sup>1</sup> The changes made for use in UHV and low temperatures are as follows. The waveforms used for the sample approach were those outlined by Besocke<sup>12</sup> with the notable exception of the amplitude of the waveforms. The waveform at room temperature has a typical peak height of 35 volts, while the peak height for low temperature operation is increased to 85 volts. This amplitude increase is used to overcome the reduction in PZT 4 piezoelectric transducer gain at lower temperatures. Also, the waveform generator used for room temperature imaging was modified to increase its current driving capacity. This was done to compensate for the increased capacitive load caused by the longer cables used in the low temperature stage. If the asymmetry of the waveform is reduced by the increased capacitive load, sample approach becomes difficult or impossible. Finally, the top portion of the outer three piezoelectric transducers were connected together using ~0.15mm tantalum foil, leaving the center, scanning piezoelectric transducer isolated. This improved the stability of the STM by reducing the excitations of the sample holder while positioned on the STM. Finally, the resonances of the sample holders were damped by machining them to be highly asymmetric.<sup>13</sup>

The ACSTM control cables and microwave cables are low temperature compatible. The control cables, *e.g.* piezoelectric transducer electrode connections, are all made with ultrasmall stainless steel coaxial cable.<sup>14</sup> These cables extend from the top of the dewar down to the eight pin UHV electrical feedthroughs. The microwave coaxial cables have stainless steel outer conductors and Cu clad BeCu center conductors separated by Teflon insulators that compensate for thermal expansion.<sup>15</sup> These cables span the same length as the control cables, but unlike the flexible control cables, the microwave cables are extremely rigid. To reduce the coupling of vibrations to the

cryogenic stage and reduce the ringing/resonating of these cables, a 25cm section of flexible microwave coaxial cable is inserted at the top of the cryostat.<sup>16</sup> This portion of the cryostat is close to room temperature and this allows the cable to remain flexible.

### C. Vertical Sample Translator

For all vertical motions a claw assembly is used to hold and to manipulate the disk-shaped beetle-style STM sample holder. This claw is mounted on a combination linear-rotary feedthrough,<sup>17</sup> which is in turn mounted on a welded bellows supported and guided on a long-stroke linear positioning table.<sup>18</sup> The rotary motion allows for the proper orientation of the sample holder ramps at the starting position for approach. The claw's cylindrical aperture corrals the sample holder during retrieval. This provides us with a large margin of error for alignment of the translator with respect to the sample during the blind pickup from the low temperature ACSTM stage. The actuation of the claw mechanism is detailed in Fig. 3, where the coaxial linear motion of the translator is used to depress the plunger that clamps and releases the opposing halves of the claw. As shown in the figure, the claw mechanism is spring loaded in two directions. This permits a range of clamping strengths as well as providing flexibility. We have found this claw design to be very versatile as well as forgiving with respect to the alignment of the sample holder at the point of pickup.

The elevator, which mates with and aligns the claw assembly, accomplishes the last 5cm of sample translation to the ACSTM. The elevator assembly shown in Fig. 4 is comprised of an OFHC Cu elevator platform, a precision stainless steel ball screw,<sup>18,19</sup> and a UHV rotary feedthrough.<sup>20</sup> The rotary feedthrough is attached to a precision stainless steel ball screw which is used to translate the elevator platform up and down.<sup>21</sup> After the sample is placed in the elevator and released by the claw, the funnel shaped bottom of the Cu elevator platform aligns and centers the sample holder automatically as it docks with the vertically mounted beetle-style ACSTM.<sup>22</sup> The preferred starting position for operation of the approach mechanism of the beetle-style ACSTM is to have the sample centered. Centering of the sample in the elevator platform also aids in the pickup of the sample by the

claw. The separate motions of the long stroke vertical translator and the elevator also protect the ACSTM from possible physical damage during blind sample transfer.

#### **D. Sample Preparation Stage**

The sample holder is lowered onto the heater stage in the room temperature UHV chamber by the vertical translator claw assembly. The heater element is then lowered on top of the sample holder and clamps the sample holder in place. The details of the sample heater stage are shown schematically in Fig. 5. The heater stage is mounted on a dual linear/rotary feedthrough.<sup>23</sup> While one linear motion centers the heater stage for sample exchange, the other clamps the heater onto the sample holder from behind. The rotary motion of the heater platform and heating element is used to orient the sample properly for ion sputtering and simultaneous or independent heating. The heating element is electrically isolated from the rest of the heater stage while the sample holder rests on an insulating alumina heater platform. This electrical isolation of the heater and sample provides a means by which the ion current to the sample can be monitored during sputtering of conducting samples. Isolation also provides us with the ability to bias the sample. The heating element used is a potted heater that can operate up to 1200K.<sup>24</sup> Higher temperatures can be obtained by replacing the potted heater with an electron beam (cathode) heater.<sup>24</sup> This electron beam heater is much smaller in size than the potted heater. To accomplish the same clamping normally provided by the potted heater, the electron beam heater is mounted in a casing similar in size to the potted heater.

Several samples can be stored in the room temperature UHV chamber using a linear motion feedthrough with two buckets which mimic the design of the sample buckets located in the sample elevators. Transfer to and from this sample tray is carried out by moving the tray under the vertical translator where the claw is then used to exchange samples. The same design is used on the long stroke horizontal translator for moving samples in UHV in and out of the load lock.

### **III. Operation**

Fig. 6 shows an example of a 750 Å x 750 Å conventional STM image of Cu(111) obtained at room temperature in UHV. The Cu(111) single-crystal was prepared by repeated Ar<sup>+</sup> ion sputtering

and annealing cycles after an initial electrochemical etching. The sample was then transferred through the chamber into the room temperature ACSTM. The image was recorded in constant current mode at a tunneling current of 0.1 nA with a bias voltage of  $-0.1\text{V}$  applied to the polycrystalline tungsten tip. The area shown in Fig. 6 includes a screw dislocation and a number of monatomic height steps. The gray scale representations in Figs. 6 and 7 depict the difference in height between successive points on a line scan. Going from left to right, a rise or a positive derivative in topography between points is depicted as bright, while a negative derivative is depicted as dark.

Shown in Fig. 7 is a  $200\text{ \AA} \times 200\text{ \AA}$  image of benzene on Cu(111) recorded at 77K in UHV. The single-crystal was prepared in the same fashion as above. Next, it was dosed at room temperature with benzene through a leak valve. The sample was then transferred to the OFHC Cu elevator where it was allowed to cool and then lowered onto the low temperature STM. The image was recorded in constant current mode at a tunneling current of 1.0 nA and a tip bias voltage of  $-0.1\text{V}$ . The area imaged includes a monatomic height step decorated with benzene molecules, two isolated clusters of benzene molecules on the upper terrace, and several vacancy defects in the Cu(111) surface on the lower terrace. Inside the largest defect, disordered benzene molecules are visible. Also apparent in the image are standing waves in the electron density as previously seen by Crommie *et al.*<sup>25</sup>

Both images were recorded with all vacuum equipment (i.e. turbo molecular pumps, mechanical pumps, and cooling water) left on. All surface preparation and analysis was performed at a background pressure in the  $10^{-10}$  torr range. During sample transfer, the background pressure remains in the  $10^{-10}$  torr range. The data recorded at 77K was taken with liquid nitrogen as the cryogen. The disturbance caused by the normal liquid nitrogen boil off posed no apparent tunnel junction stability problems. The application of the high frequency modulation in UHV and at 77K again caused no heating or outgasing problems. The performance of the ACSTM in this UHV, low temperature configuration was comparable to that of our previously reported design.<sup>1</sup> Simultaneous

conventional and ACSTM images were taken on the benzene/Cu(111) surface with no degradation of the conventional STM image.

#### **IV. Conclusions and Prospects**

We have found this low temperature, UHV ACSTM to be very stable and reliable. We will use this instrument to attempt to record rotational spectra of single molecules and other atomic scale spectroscopies as described in Ref. 2.

### **Acknowledgments**

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5. This mirror is particularly useful for the cryogenic ACSTM, but its use must be limited to prevent unnecessary radiative heating.
6. P. S. Weiss and D. M. Eigler, *Nanosources and Manipulation of Atoms Under High Fields and Temperatures: Applications*, 213 (Kluwer Academic Publishers, Netherlands, 1993).
7. Insulator Seal Incorporated, Hayward, CA.
8. Newport Corp., Fountain Valley, CA.
9. Barry Controls, Watertown, MA.
10. DuxSeal<sup>®</sup> works fine and is easily removed and replaced.
11. National Electrostatics Corporation, Middleton, WI.
12. K. Besocke, *Surf. Sci.* **181**, 145 (1987); J. Frohn, J. F. Wolf, K. Besocke, and M. Teske, *Rev. Sci. Instrum.* **60**, 1200 (1989).
13. The sample holder's symmetry is reduced by milling notches in the back side of the holder in random locations. This does not change the normal operation of the STM. It merely lowers the resonance amplitude.
14. Cooner Wire, Chatsworth, CA.
15. Isocore Coaxial Cable, Rogers Corporation, Chandler, AZ.
16. W.L. Gore & Associates, Electronic Products Division, Newark, DE.
17. Huntington Laboratories, Inc., Mountain View, CA.
18. Linear Industries, Monrovia, CA.

19. Note that the ball screw is manufactured with nylon bearing deflectors which we have replaced with Cu replicas machined for use in UHV.
20. Varian Vacuum Products, Lexington, MA.
21. The ball screw has been replaced in other versions of this elevator assembly by a welded bellows linear feedthrough. This provides comparable performance to the ball screw assembly and is commercially available without the need for modification.
22. This means of sample centering reduces the number of times that the vertical translator must be used. This reduces the thermal load on the cryostat and maintains all parts of the low temperature ACSTM at cryogenic temperatures after the sample is dropped off by the vertical translator.
23. MDC Vacuum Products Corporation, Hayward, CA.
24. Spectra-Mat, Watsonville, CA.
25. M. F. Crommie, C. P. Lutz and D. M. Eigler, *Nature*. **363**, 524 (1993).

**FIGURE CAPTIONS**

**Fig. 1.** Schematic view of the ACSTM chambers, the vibration isolation platform, and the two ultrahigh vacuum ACSTMs — one operates at room temperature (RT ACSTM) and the other at low temperature (LT ACSTM). The system is supported on a pneumatic laser table (T) and the ultrahigh vacuum and low temperature chambers are decoupled by a welded bellows (B) and independently mounted on vibration isolation platforms (VIP). The sample claw (C), vertical translator (VT), and horizontal translator (HT) are used to transfer samples between the sample preparation/analysis chamber, the load lock, and the ACSTMs.

**Fig. 2.** Schematic cross section of the surface preparation/analysis chamber and the load lock. The claws are used to transfer samples between the heating/sputtering stage, sample tray, and horizontal translators.

**Fig. 3.** A schematic of the claw. The actuation of the claw mechanism is accomplished with the coaxial linear motion of the translator. The linear plunger of the translator (A) presses against the stainless steel ball bearings (B) and compresses the springs (C) to the point where the linear plunger of the claw (D) begins to move forward thereby closing the jaws of the claw. As shown, the claw mechanism is spring loaded in two directions permitting a range of clamping strengths. The claw's cylindrical jaws (E) have tapered knife edges that aide in sample alignment during pickup. The bearing wheel keying mechanism (F) is mounted on the outer shaft of the claw. While the bearing wheels key into the alignment sleeve and remain stationary, the claw assembly is free to rotate.

**Fig. 4.** The elevator assembly shown is comprised of a Cu elevator platform (A), a precision stainless steel ball screw (B), and a UHV rotary feedthrough (C). The sample is placed into the elevator and released by the claw (D). The funnel shaped bottom of the Cu elevator platform aligns and centers the sample holder disk (E) as it is lowered on to the vertically mounted beetle-style STM (F).

**Fig. 5.** The heating stage is mounted on a dual linear/rotary feedthrough. While one of the linear motions (A) centers the heater for sample exchange, the other (B) clamps and lowers the heating element (C) onto the back of the sample holder (D). The rotary motion (E) of the heating stage is used to orient the sample holder for ion sputtering. The heating element is electrically isolated from the rest of the heater stage by an alumina shaft (F) while the sample rests on an alumina pan (G). This is done to allow measurement of the ion current to the sample during sputtering.

**Fig. 6.** A  $750 \text{ \AA} \times 750 \text{ \AA}$  STM image of Cu(111) recorded at room temperature in UHV. The image was recorded in constant current mode at a tunneling current of 0.1 nA and a tip bias voltage of  $-0.1 \text{ V}$ . The area imaged includes a screw dislocation and a number of monatomic height steps. See the text for a description of the rendering used.

**Fig. 7.** A  $200 \text{ \AA} \times 200 \text{ \AA}$  STM image of benzene on Cu(111) recorded at low temperature,  $T=77 \text{ K}$ , in UHV. The image was recorded in constant current mode at a tunneling current of 1.0 nA and a tip bias voltage of  $-0.1 \text{ V}$ . The area imaged includes a monatomic height step decorated with benzene molecules, two isolated clusters of benzene molecules on the upper terrace, and several defects on the lower terrace. Inside the largest defect, disordered benzene molecules are visible. Also apparent in the image are standing waves in the electron density. See the text for a description of the rendering used.

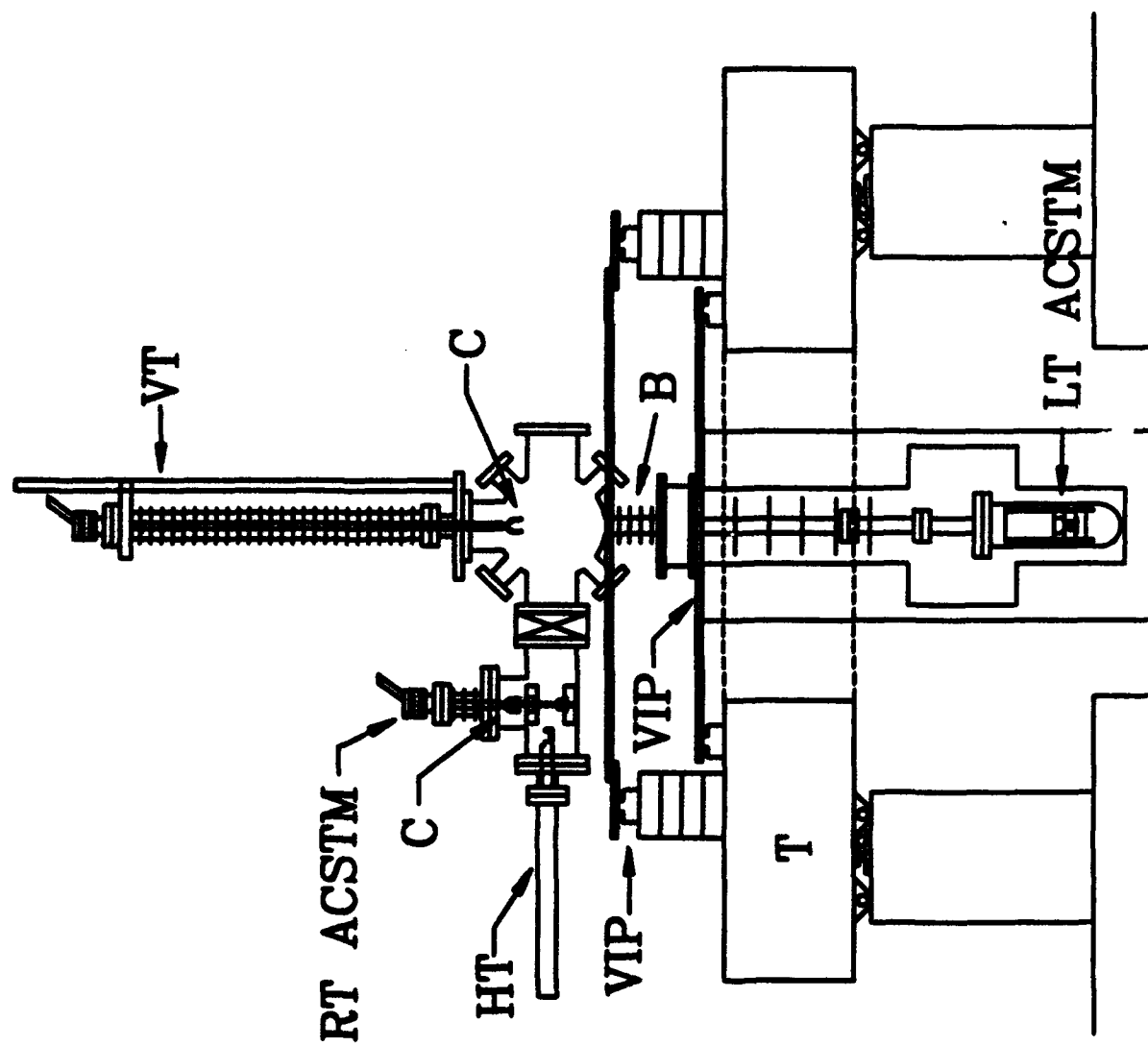


Fig. 1

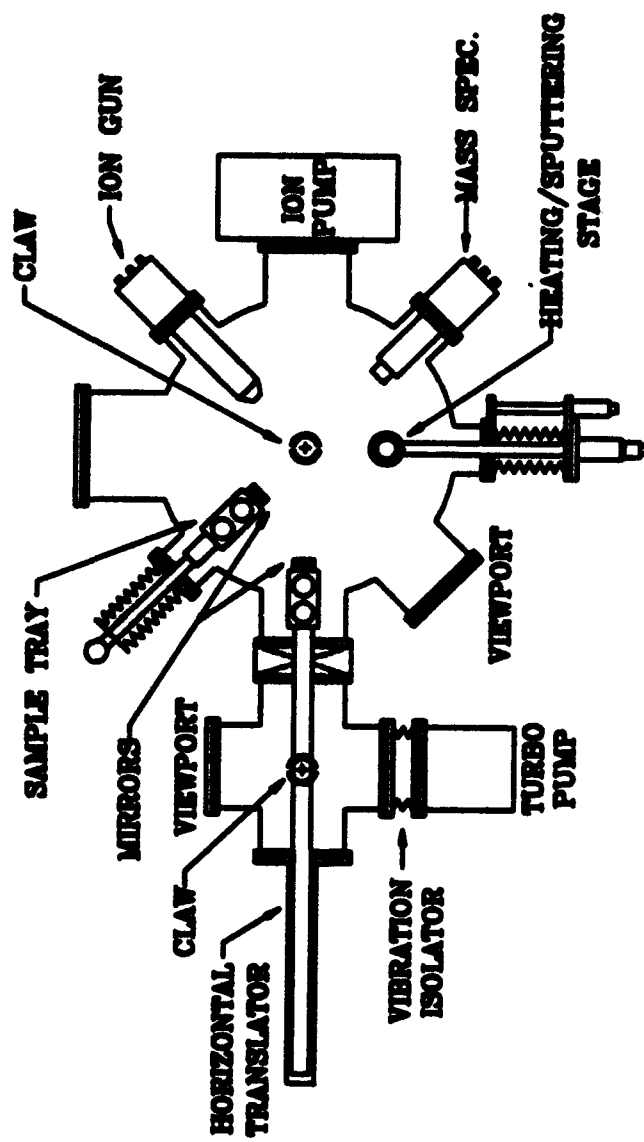


Fig. 2

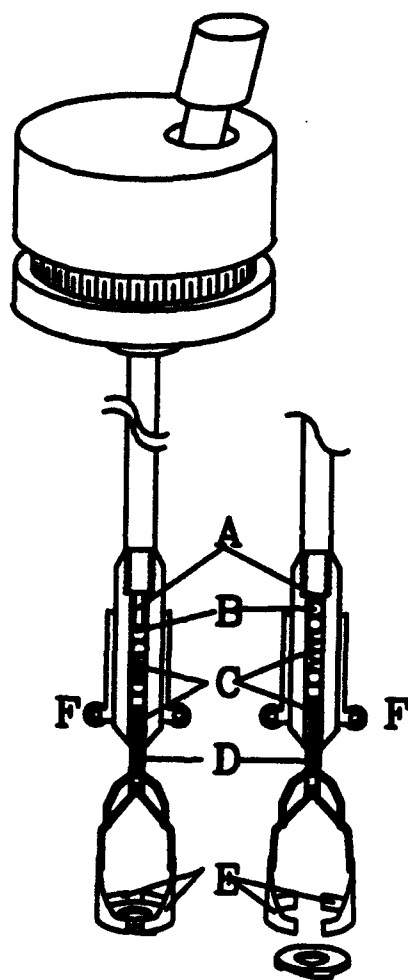
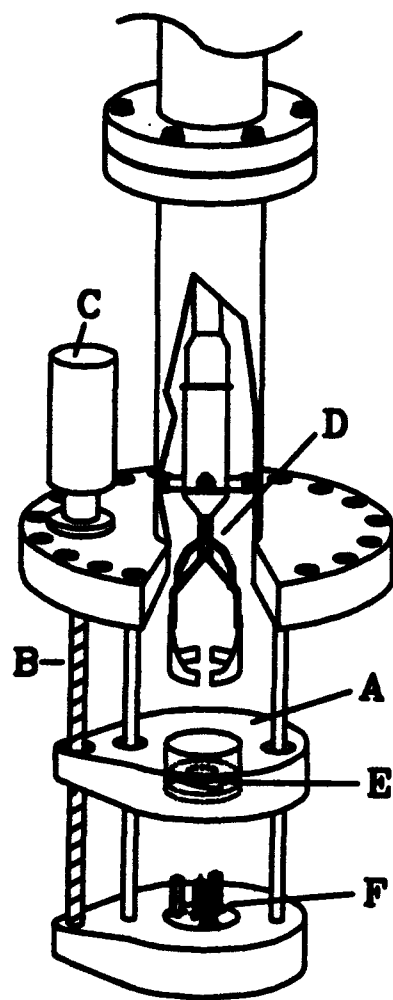


Fig. 3



**Fig. 4**

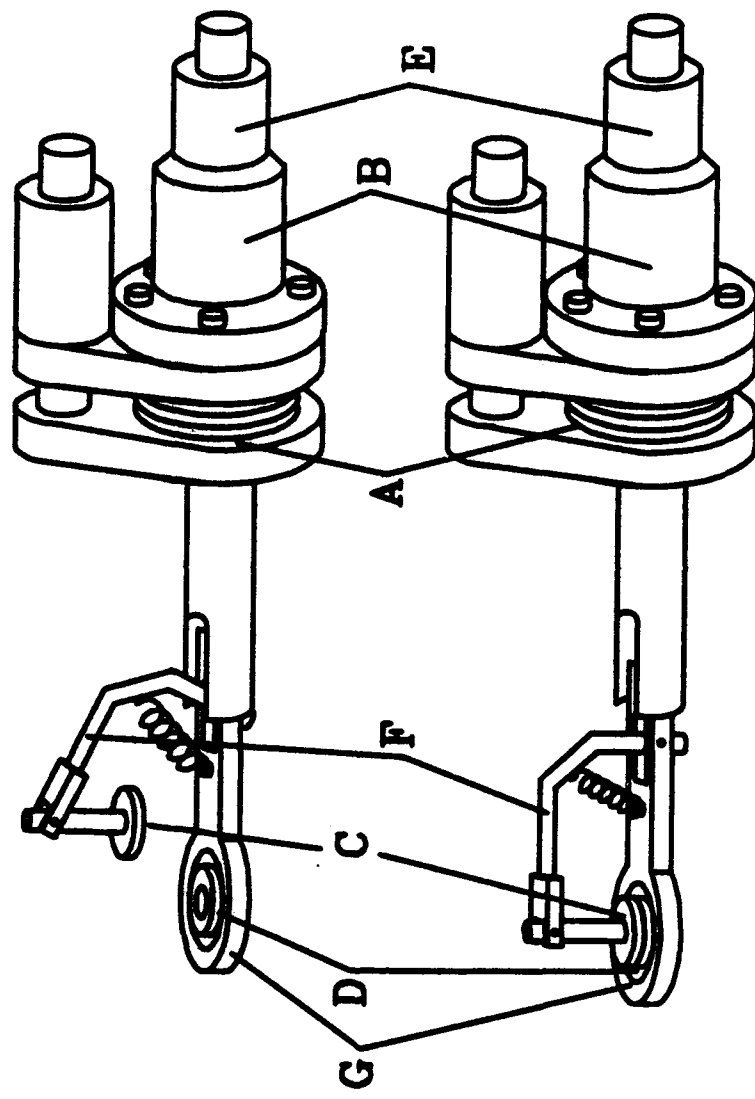
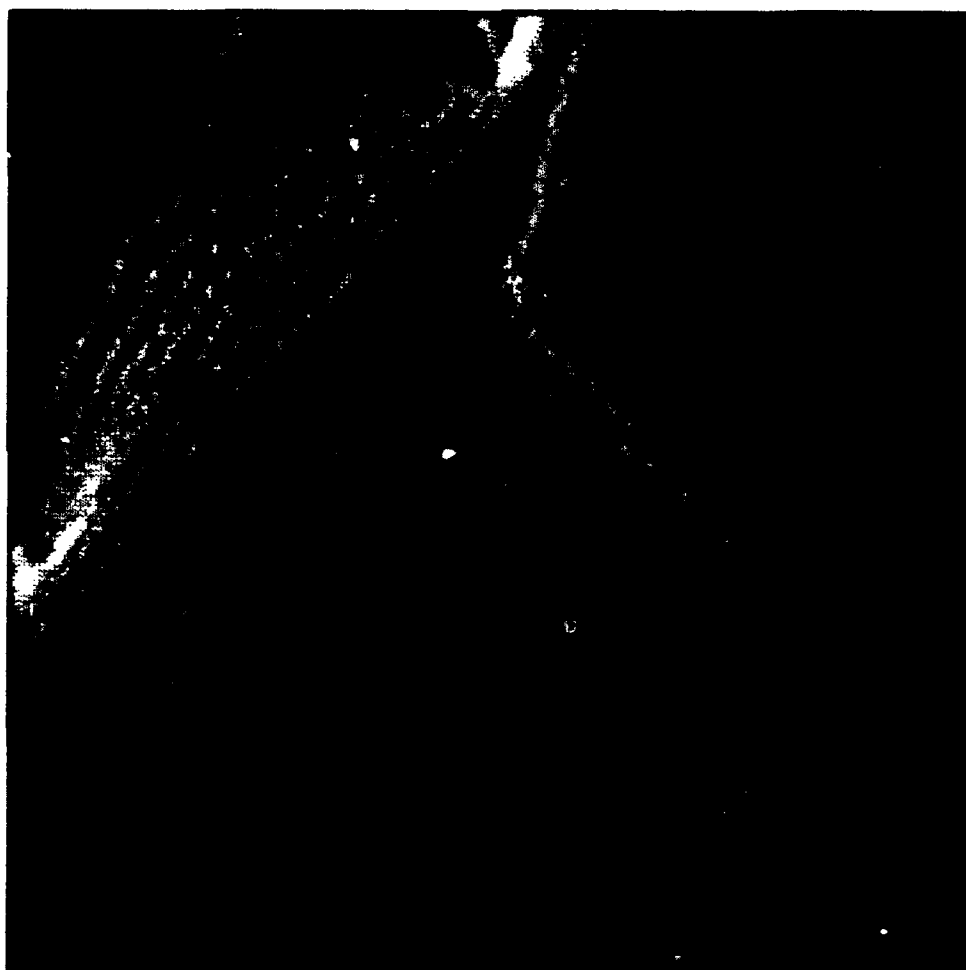


Fig. 5

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Fig. 6 UP<sup>+</sup>



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Fig. 7

UP<sup>1</sup>

